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मानक

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Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

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IS 326-21 (2001): Methods of Sampling and Test for Natural and Sythetic Perfumery Materials, Part 21: Determination of Water Content - Karl Fischer Method [PCD 18: Natural and Synthetic Fragrance Materials]



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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

प्राकृतिक और संश्लिष्ट सुगन्ध सामग्रियों के नमूने लेने
की परीक्षण पद्धतियाँ

भाग 21 पानी की मात्रा ज्ञात करना — कार्ल फिशर विधि

Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL
AND SYNTHETIC PERFUMERY MATERIALS

PART 21 DETERMINATION OF WATER CONTENT — KARL FISCHER METHOD

ICS 71.100.60

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard (Part 21) which is identical with ISO 11021:1999 'Essential oils — Determination of water content — Karl Fischer method' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of Natural and Synthetic Fragrance Materials Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO Standard has been approved as suitable for publication as Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear, they should be read as 'Indian Standard'.
- b) Comma (,) has been used as a decimal marker, while in Indian Standards, the current practice is to use a full point (.) as the decimal marker.

In this adopted standard, reference appears to one International Standard for which Indian Standard also exists. The corresponding Indian Standard which is to be substituted in its place are listed below along with its degree of equivalence for the edition indicated:

<i>International Standard</i>	<i>Corresponding Indian Standard</i>	<i>Degree of Equivalence</i>
ISO 760:1978 Determination of water — Karl Fischer method (General method)	IS 2362 : 1993 Determination of water by the Karl Fischer method (<i>first revision</i>)	Technically equivalent

For tropical countries like India, the standard temperature and the relative humidity shall be taken as $27 \pm 2^{\circ}\text{C}$ and 65 ± 5 percent respectively.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

*Indian Standard***METHODS OF SAMPLING AND TEST FOR NATURAL
AND SYNTHETIC PERFUMERY MATERIALS****PART 21 DETERMINATION OF WATER CONTENT — KARL FISCHER METHOD****1 Scope**

This International Standard specifies a method for the determination of the water content of essential oils by the Karl Fischer method.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 760, *Determination of water content — Karl Fischer method (General method)*.

3 Terms and definitions

For the purposes of this International Standard, the following term and definition apply.

3.1**water content**

amount of water present in the essential oil considered, determined in accordance with the procedure specified in this International Standard

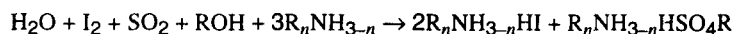
NOTE Water content is expressed as a mass fraction in percent [formerly designated as % (m/m)].

4 Principle

The water present in a test portion is absorbed with dried methanol. The water is allowed to react with the Karl Fischer reagent without pyridine, previously standardized by titration using a Karl Fischer apparatus. The endpoint of the reaction is obtained by an electrometric method.

5 Reactions

During the determination of water according to the Karl Fischer method, the water present in the sample reacts, in the presence of an amine and an alcohol, with iodine and sulfur dioxide:



where R is an alkyl or alkoxy group.

The endpoint of the reaction is obtained electrometrically by a surplus of iodine.

6 Reagents and materials

Use only reagents of recognized analytical quality and distilled or demineralized water or water of equivalent purity.

6.1 Methanol, containing not more than 0,05 % of water by mass. If the reagent contains more than this quantity of water, dry it by distillation in an inert atmosphere (nitrogen, helium etc.) with magnesium turnings activated with iodine. Collect the distillate in a flask protected from atmospheric moisture by means of a guard tube with a desiccant. Alternatively, use 2-methoxyethanol (6.2).

6.2 2-Methoxyethanol (ethylene glycol monomethyl ether or methylcellosolve), containing not more than 0,05 % of water by mass. If the reagent contains more than this quantity of water, dry it by distillation, rejecting the first few millilitres of the distillate which contains water.

6.3 Karl Fischer reagent,¹⁾ free from pyridine, having a titre lower than 4,0 mg/ml; one or two component system. The titre shall be determined daily.

If carbonyl compounds are present, an adapted Karl Fischer reagent shall be used.

6.4 Tartaric acid, dipotassium salt ($C_4H_4O_6K_2 \cdot \frac{1}{2}H_2O$), or **water**.

7 Apparatus

See ISO 760.

All the glassware used shall be previously dried for 30 min in an oven maintained at approximately 130 °C, then stored in a desiccator containing a desiccant then allowed to cool to room temperature.

The automatic burettes should not be heated in an oven. They should be dried with absolute ethanol or acetone and a dry air stream.

Usual laboratory apparatus and, in particular, the following.

7.1 Titration apparatus

There is commercially available apparatus for the automatic determination of the water content according to the Karl Fischer method, which consists of the items given in 7.1.1 to 7.1.5.

7.1.1 Endpoint indicator, for titration according to the electrometric method.

7.1.2 Platinum double electrode.

7.1.3 Magnetic stirrer, fitted with a stirring rod covered with polytetrafluoroethylene, operating at a rotational frequency of 150 r/min to 300 r/min.

7.1.4 Titration vessel, of approximately 200 ml capacity, with at least three ground glass sockets.

Connect one of the ground glass sockets to the automatic burette. Place the platinum double electrode in the automatic burette, and the platinum double electrode in the second one, and use the third one to add the reagents and the sample. A discharge tap at the bottom of the vessel is an advantage. Connect it to the vessel with a capillary tube having a volume of not more than 0,5 ml. Close all ventilation sockets in the titration vessel either with drying tubes which have been charged with an effective drying agent or molecular sieves, or with drying receivers filled with the reagent (6.3) in order to avoid the influence of moisture from the air.

¹⁾ Suitable products are commercially available.

7.1.5 Automatic burette, of 25 ml capacity, graduated in intervals of 0,05 ml, and reagent bottle and drying tubes.

The minimum interval of the burette depends on data from the manufacturer (e.g. 0,02 ml).

Protect the reagent bottle, the burette and the Karl Fischer reagent (6.3) from moisture and light.

7.2 Oven, regulated at $130\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$, to dry the glassware.

8 Sampling

A recommended sampling method is given in ISO 212.

NOTE Attention is drawn to the fact that the method given in ISO 356 cannot be used to prepare the test samples as it specifies that the sample has to be dried.

9 Procedure

9.1 Preparation of the titration apparatus

Prepare the titration apparatus (7.1) in accordance with the manufacturer's instructions and lubricate every joint. The air-tightness of the titration vessel is of special importance.

Place 25 ml of the dried methanol (6.1) or of the 2-methoxyethanol (6.2) in the titration vessel (7.1.4). Be sure that the metallic parts of the platinum double electrode (7.1.2) are completely immersed. Start the automatic titration (7.1.3).

Add Karl Fischer reagent (6.3) to enable a reaction to take place with the traces of water in the methanol. The titration and the recognition of the endpoint is controlled automatically by the apparatus.

9.2 Standardization of the Karl Fischer reagent

Prepare the titration apparatus as indicated in 9.1.

Weigh, to the nearest 0,000 1 g, approximately 0,300 g of the potassium bitartrate (6.4). Introduce quantitatively and as fast as possible, the mass (m_1) of the bitartrate to the titration vessel (7.1.4).

Titrate the known amount of water introduced in the vessel by the dipotassium salt of tartaric acid, with the Karl Fischer reagent to perform the standardization. The titration and the endpoint are controlled automatically by the apparatus.

Record the volume (V_1) of the reagent used with the bitartrate.

Standardization of the reagent may also be carried out with a known mass of pure water (m_2), approximately 0,040 g, measured to the nearest 0,000 1 g.

Record the volume (V_2) of the reagent used with the water.

Carry out several titrations without changing the titration vessel. Use the arithmetic mean of the results for the calculation of the water equivalent of the reagent (10.1).

9.3 Determination

Prepare the titration apparatus as indicated in 9.1.

Depending on the expected water content of the sample, take a portion (V_0 ml) of the sample (10,0 ml if the water content is $<3\%$ or 25,0 ml if the water content is $<1\%$) and place it very quickly in the titration vessel at a temperature below $25\text{ }^{\circ}\text{C}$, as slurring and dissolution may occur.

Start the automatic titration while stirring with the magnetic stirrer (7.1.3). The titration and the recognition of the endpoint are controlled automatically by the apparatus.

Record the volume (V_3) of the reagent used.

Carry out three titrations on each sample and use the arithmetic mean of the results for the calculation (10.2).

10 Expression of results

10.1 Water equivalent of the Karl Fischer reagent

The calculation of the water equivalent, T , of the Karl Fischer reagent (6.3), expressed in milligrams of water per millilitre of reagent, is given by one of the formulae:

$$T = \frac{m_1}{V_1} \times \frac{0,5 \times 18}{235,28} \quad \text{or} \quad T = \frac{m_2}{V_2}$$

where

m_1 is the mass, in milligrams, of the dipotassium salt of tartaric acid (6.4) introduced when this reagent is used for standardization (9.2);

V_1 is the volume, in millilitres, of the reagent (6.3) used for the standardization;

0,5 is the number of water moles per mole of dipotassium salt of tartaric acid;

18 is the relative molecular mass of water;

235,28 is the relative molecular mass of dipotassium salt of tartaric acid;

m_2 is the mass, in milligrams, of the known pure water (6.4), if water is used for standardization;

V_2 is the volume, in millilitres, of the Karl Fischer reagent (6.3) used for the standardization.

10.2 Water content of the essential oil

The water content of the essential oil, W_w , expressed as a mass fraction in percent, is given by the formula:

$$W_w = \frac{V_3 \times T}{V_0 \times \rho \times 10}$$

where

V_0 is the volume, in millilitres, of the test portion (9.3);

ρ is the density of the sample, in grams per millilitre, at 20 °C;

V_3 is the volume, in millilitres, of the Karl Fischer reagent (6.3) used for the determination (9.3);

T is the water equivalent, in milligrams of water per millilitre of the Karl Fischer reagent (6.3), calculated in accordance with 10.1.

To calculate the density of the essential oil, multiply the value obtained for the relative density by the density of water at 20 °C, that is 0,998 23 g/ml.

11 Test report

The test report shall specify:

- the method used,
- the test result obtained;
- any operating details not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the test result.

The test report shall include all information required for the complete identification of the sample.

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :

	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110 002	{ 323 76 17 323 38 41
Eastern : 1/14 C.I.T. Scheme VII M, V. I. P. Road, Kankurgachi KOLKATA 700 054	{ 337 84 99, 337 85 61 337 86 26, 337 91 20
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160 022	{ 60 38 43 60 20 25
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600 113	{ 254 12 16, 254 14 42 254 25 19, 254 13 15
Western : Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400 093	{ 832 92 95, 832 78 58 832 78 91, 832 78 92
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